

# UNITED STATES ENVIRONMENTAL PROTECTION AGENCY WASHINGTON, D.C. 20460

OFFICE OF PREVENTION, PESTICIDES AND TOXIC SUBSTANCES

August 17, 2007

**MEMORANDUM** 

SUBJECT: Review of "Measurement of Transfer of Pyrethrin and Piperonyl Butoxide

Residues from Vinyl or Carpet Flooring Treated with a Fogger Formulation to

Glove Coupons Following Multiple Presses"

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Attached is a review of a study conducted to determine the transfer of pyrethrin (PY) and piperonyl butoxide (PBO) residue from formulation-treated vinyl and carpet flooring sections to glove coupons following multiple presses. The study was submitted by the Non-Dietary Exposure Task Force. The study review was conducted by Versar, Inc. A secondary review was conducted by HED.

## **Executive Summary**

The purpose of the study was to determine the effect of applied force on residue transfer of formulated pyrethrin (PY) and piperonyl butoxide (PBO) after a single application of an unidentified pre-fill batch fogger formulation containing 0.77% pyrethrin and 1.64% piperonyl butoxide as the active ingredients. Simulated Residential Rooms (SRRs) and sprayboom application equipment were used to simulate a residential fogger release scenario. The sprayboom apparatus was used to generate a target deposition of 3.96  $\mu$ g/cm² for PY and 7.87  $\mu$ g/cm² for PBO. Deposition rates were based on results of indoor PY and PBO total release fogger deposition studies. Total deposition was measure using deposition coupons collected 3.5 hours application of the test material.

To measure transfer of residue, cotton glove coupons were attached to wooden blocks (10" x 10"). The cotton glove coupon/wood block was placed on a designated area of the vinyl or carpet flooring section and then an 8 kg weight was immediately placed on top of the cotton glove coupon/wood block for a duration of 20 seconds. For the first round of presses, a new area of treated vinyl or carpet flooring section was used. Three replicates were used for each number of presses (2, 4, 8, 16, or 32). Following the first round of presses, a new cotton glove coupon was placed on the same area of each previously used flooring section for a repeat of exposure. This process was repeated three additional times for each section of flooring for a total of four rounds. The cotton gloves and deposition coupons were extracted and then analyzed using GC/MS.

The percent of residue on the cotton glove coupons after contact with treated vinyl and carpet flooring surfaces was calculated as the ratio of the amount of residue present on the glove coupons divided by the average corrected residue found on the alpha cellulose coupons (5.73  $\mu g/cm^2$  for PY and 13.1  $\mu g/cm^2$  for PBO).

For the residue transferred from vinyl flooring to cotton glove coupons, the percent of application for PY and PBO per round of presses increases with an increase in the number of presses (from 2 to 16) and then decreases at 32 presses. For example, for Round 1, the percent of PY and PBO after 2 presses is 0.645% and 0.404%, after 16 presses is 3.15% and 2.12%, and after 32 presses is 1.61% and 1.07%, respectively. The percent of application for PY and PBO per press decreases with an increase in the number of presses. For example, for Round 1, the percent of PY and PBO after 2 presses is 0.323% and 0.202% and after 32 presses is 0.050% and 0.033%, respectively.

For the residue transferred from carpet flooring to cotton glove coupons, the percent of application for PY and PBO per round of presses increases with an increase in the number of presses. For example, for Round 1, the percent of PY and PBO after 2 presses is 1.34% and 1.26% and after 32 presses is 14.4% and 13.0%, respectively. The percent of application for PY and PBO per press initially increases from 2 to 4 presses or 2 to 8 presses, and then decreases to 32 presses. For example, for Round 1, the percent of PY and PBO after 2 presses is 0.670% and 0.628%, after 4 presses is 0.729% and 0.707%, and after 32 presses is 0.450% and 0.406%, respectively.

Results of the study indicate that for vinyl flooring, per round of presses, there appears to be a limit to the amount of residue that is transferred with an increase in the number of contacts with the treated flooring. Increasing the number of contacts initially increases the percent of residue transferred, but at a certain point, a maximum loading is reached. However, per press, the amount of residue transferred continues to decrease with an increase in the number of presses, most likely because there is less residue available after each press.

Results of the study indicate that for carpet flooring there is a somewhat different pattern. Per round of presses, the percent of residue continues to increase with an increase in the number of contacts. There does not appear to be a point at which a maximum loading is reached. The percent of residue transferred per press for carpet flooring increases initially and then decreases with an increase in the number of presses.

When comparing carpet versus vinyl flooring, the percent transfer per round and also per press is higher for carpet than it is for vinyl flooring. It appears that residues are more available from carpet than from vinyl flooring, and exhibit slightly different patterns with an increase in the number of contacts.

The requirements for this study were specified by the U.S. Environmental Protection Agency's (U.S. EPA) OPPT Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines. The relevant guidelines and the protocol provided along with the study were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set for the in the protocol and guidelines. The data are of sufficient scientific quality to be used to determine exposure.

**Reviewers:** Teri Schaeffer **Date:** March 11, 2004

**STUDY TYPE:** Active Transfer; Vinyl and Carpet

**TEST MATERIAL:** An unidentified pre-fill batch fogger formulation containing 0.77% pyrethrin (CAS no.

8003-34-7) and 1.64% piperonyl butoxide (CAS no. 51-03-6) as the active ingredients

was used.

**SYNONYMS:** Pyrethrin (PY) and Piperonyl Butoxide (PBO)

**CITATION:** Author: Sami Selim, Ph.D.

Study Director: Robert E. Rogers, Ph.D., D.A.B.T.

Title: Measurement of Transfer of Pyrethrin and Piperonyl Butoxide

Residues from Vinyl or Carpet Flooring Treated with a Fogger

Formulation to Glove Coupons Following Multiple Presses

Report Date: August 25, 2002

Testing Facility: Toxcon Health Sciences Research Centre Inc.

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Analytical Facility: Enviro-Test Laboratories/XENOS Division

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Identifying Codes: Toxcon Study Number: 00-034-PY01

Xenos Project Number: XEN00-33

Unpublished

**SPONSOR:** Non-Dietary Exposure Task Force

## **EXECUTIVE SUMMARY:**

This report reviews the study "Measurement of Transfer of Pyrethrin and Piperonyl Butoxide Residues from Vinyl or Carpet Flooring Treated with a Fogger Formulation to Glove Coupons Following Multiple Presses" submitted by the Non-Dietary Exposure Task Force. The purpose of the study was to first determine the degree of transfer of pyrethrin (PY) and piperonyl butoxide (PBO) residue from previously untouched areas of treated vinyl and carpet flooring after a single application of an unidentified pre-fill batch fogger formulation containing 0.77% PY and 1.64% PBO as the active ingredients. Secondly, the study was to determine transfer of PY and PBO residue from the same area of treated vinyl and carpet flooring as a function of multiple contacts.

A test room was prepared with wooden platforms placed in the center of the room. Vinyl and carpet flooring sections were placed on the platforms along with deposition coupons. A single application of the test product was applied using one sprayboom run to obtain a desired deposition of  $3.96~\mu g/cm2$  of PY and  $7.87~\mu g/cm2$  of PBO onto the vinyl and carpet flooring surfaces. During the application, and for three hours after the application, the ventilation system in the room was turned off (with the dampers closed) to allow for deposition of airborne formulation onto the test surfaces. After the three hours, the dampers were opened for a 30 minute drying period. Following the 30 minute drying period the degree of transfer of PY and PBO from treated vinyl and carpet flooring sections was measured using cotton glove coupons following: (1) a single press using an 8 kg force placed on top of the glove coupon/wooden block and (2) multiple presses (2, 4, 8, 16, or 32 consecutive presses) using an 8 kg force on top of the glove coupon/wooden block.

The achieved deposition rate was calculated by Versar to be 145% of the target deposition rate for PY and 167% of the target deposition rate for PBO. For vinyl flooring, the average residues for the different number of presses for

each of the four rounds ranged from  $0.019~\mu g/cm2 \pm 0.004~\mu g/cm2$  (4th round, after 2 presses) to  $0.180~\mu g/cm2 \pm 0.050~\mu g/cm2$  (1st round, after 16 presses) for PY and from  $0.029~\mu g/cm2 \pm 0.006~\mu g/cm2$  (4th round, after 2 presses) to  $0.278~\mu g/cm2 \pm 0.067~\mu g/cm2$  (1st round, after 16 presses) for PBO.

Cotton glove coupons generated for carpet flooring, showed average residues for the different number of presses for each of the four rounds ranging from  $0.041~\mu g/cm2 \pm 0.008~\mu g/cm2$  (4th round, after 2 presses) to  $0.825~\mu g/cm2 \pm 0.065~\mu g/cm2$  (1st round, after 32 presses) for PY and from  $0.091~\mu g/cm2 \pm 0.013~\mu g/cm2$  (4th round, after 2 presses) to  $1.073~\mu g/cm2 \pm 0.133~\mu g/cm2$  (1st round, after 32 presses) for PBO.

The transfer of PY and PBO residues from treated vinyl and carpet flooring to cotton glove coupons following multiple presses on new or the same treated area was measured. The results of this study indicated that for vinyl and carpet flooring, the per press residues of both PY and PBO transferred from the flooring surface to the cotton glove coupons decreased as the number of presses increased. For vinyl, the percent of PY transferred to the cotton glove coupons was greater than the percent of PBO transferred. For carpet, the percent of PY and PBO transferred to the cotton glove coupons was similar.

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines. However, certain issues of concern were noted:

- A specific application rate was not provided in the Study Report. Application was based on a target deposition rate determined in another study.
- The test product was not identified and a label was not provided.
- Calibration procedures for the application equipment were not provided in the Study Report.
- Only one sampling interval was intended to collect samples. However, there was insufficient time to complete the experiment the same day as the application and the remainder of the samples were collected on the day after the application.
- The requirement for the transfer of collected samples to freezer storage within the 3-hour period was overlooked and all samples were placed into freezer storage at one time at the end of the experiment (the day after the application).
- Only one field fortification level with duplicate field fortified control samples were prepared for alpha cellulose and cotton glove coupons.
- Alpha cellulose coupons used to determine the application rate of the sprayboom were analyzed as part of another study (Xenos Report XEN00-32; Vol. 8). However, alpha cellulose coupon QC samples were prepared and analyzed specifically for this study. The field fortification recoveries for both PY and PBO were below 90% and the alpha cellulose coupon residue data were corrected (by Versar), accordingly.

# **COMPLIANCE:**

A signed and dated Data Confidentiality statement was provided. A signed and dated GLP Compliance Statement was provided, however, it was noted that this study was not performed according to the US EPA FIFRA Good Laboratory Practice Regulations currently in effect (40 CFR, Part 160). It was also noted that the data collection and study conduct were performed "in the spirit of GLP". A Quality Assurance statement was provided in the Xenos Laboratories, Inc. Analytical Phase Report.

## GUIDELINE OR PROTOCOL FOLLOWED:

The study was conducted following Xenos and Toxcon Standard Operating Procedures and the protocol of the Non-Dietary Exposure Task Force (Toxcon Protocol No. 00-034-PY01).

#### I. MATERIALS AND METHODS

#### A. Materials:

#### 1. Test Material:

Formulation: An unidentified pre-fill batch fogger formulation containing 0.77% PY and

1.64% PBO as the active ingredients.

Batch # formulation: LPB47000a

Formulation guarantee: McLaughlin Gormley King Company (MGK) Certificate of Analysis stated that

the test substance contained 0.77% total pyrethrins and 1.54% PBO. The

analysis was dated August 17, 2000.

CAS #(s): Pyrethrins: 8003-34-7

PBO: 51-03-6

Other Relevant Information: Toxcon ID No.: PY01 T006; MGK is the manufacturer of the test product.

## 2. Relevance of Test Material to Proposed Formulation(s):

The test product used for this study was a pre-fill batch formulation similar to that for an indoor fogger formulation developed by the McLaughlin Gormley King Company (MGK) intended for use in residential buildings. The name and label for this test product was not provided with the study.

## **B.** Study Design:

There were no reported amendments to the study protocol. There were four deviations from the study protocol and one deviation from the SOP reported. The four protocol deviations included: (1) there was insufficient time to complete the experiment the same day as the application; (2) the transfer of the collected samples to freezer storage within the 3-hour period required was overlooked and all samples were placed into freezer storage at one time at the end of the experiment; (3) sample dates for all the glove coupons samples collected on September 8, 2000 (day after the application) were indicated incorrectly as September 7, 2000 on the sample labels; and (4) recoveries for one of two laboratory fortified glove coupon samples analyzed in sets X003307, X003310, X003311, X003313 and X003314 did not fall within the acceptable range of 70% to 120%. The Study Director did not indicate whether or not the first three deviations would have an impact on the study results. The SOP deviation involved the demonstrated R2 values of 0.9733 (PYI) and 0.9739 (PBO) which were less than the SOP criteria of being equal to or greater than 0.98. The Study Director stated that this was not expected to impact the study.

## 1. Site Description:

Test locations:

Two test rooms, referred to as simulated residential rooms, were located at the

Toxcon Health Sciences Research Centre in Edmonton, Alberta, Canada. The rooms were prepared according to Toxcon SOP No. E-025: Preparation of Test

Rooms Prior to an Experiment.

Meteorological Data: Target test room conditions prior to application included an air exchange rate of

 $0.6 \pm 0.1$  air change per hour (ACH), a temperature of  $72 \pm 4^{\circ}F$  and a relative

humidity of  $50 \pm 10\%$ .

Ventilation/Air-Filtration: The ventilation system for the spray room was turned off during application and

for three hours after the application (with dampers closed). The dampers were opened after the three hours and for a 30 minute drying period, the room conditions were adjusted to reach the conditions prior to application.

## 2. Surface(s) Monitored:

Room(s) Monitored: Two test rooms, referred to as Simulated Residential Rooms (SRRs), were

utilized in this study. One test room contained the application equipment (sprayboom) and the second room was an untreated room used to perform the

press procedure.

Room Size(s): The dimensions of both the spray room and the untreated room were 16 ft x 16 ft

x 8 ft. Six wooden platforms (40" x 40" each) were placed in the center of the

spray room.

Types of Surface(s): Vinyl and carpet flooring

Surface Characteristics: Sections of vinyl and carpet flooring were pinned onto sheets of plastic-covered

plywood attached to the top of six wooden platforms. The vinyl and carpet flooring specifications were provided in the protocol. The vinyl flooring was made by DOMCO Inc. and featured a no-wax vinyl finish. The carpet was manufactured by KRAUS with the product name "Lasting Pleasure II." The carpet was made of saxony cut pile (100 BCF nylon) and was pre-treated with

Scotchgard.

Areas sprayed and sampled: A total of 33 vinyl flooring sections and 33 carpet flooring sections, cut into 12"

x 8" sections, were pinned onto sheets of plastic-covered plywood attached to the top of six 40" x 40" wooden platforms (A total of 15 vinyl flooring sections and 15 carpet sections were used for this study). These flooring sections were treated along with 24 deposition coupons (3" x 3"). A diagram of the deposition coupons, vinyl and carpet flooring layout was provided on page 34 of the Study

Report.

The surfaces monitored in this study were relevant to the proposed uses for this

formulated product.

Other products used: N/A

3. Physical State of Formulation as Applied : Fogger

## 4. Application Rates and Regimes:

Application Equipment: Sprayboom

Application Regime: One sprayboom run conducted in one Simulated Residential Room.

Application rate(s): An application rate was not provided in the Study Report. The application was

based on the desired deposition rate of the test product onto the vinyl and carpet flooring. For PY, the desired deposition rate was 3.96  $\mu g/cm2$  and for PBO, the desired deposition rate was 7.87  $\mu g/cm2$ . Deposition rates were based on results of indoor PY and PBO total release fogger deposition studies. The sprayboom nozzle sweep speed required to obtain the desired deposition was calculated using the following equation: U = [(Qt)(Fa)(k1)/(R)(n)(d)(10-6), where U is the sprayboom nozzle sweep speed (cm/s),Qt is the nozzle output rate (g/s), Fa is the fraction of pyrethrin in the formulation, R is the target deposition rate of PY ( $\mu g/cm2$ ), d is a fixed value representing the distance between nozzles (71.2 cm), n is the number of nozzles (5), and k1 is a correction factor to account for formulation that is sprayed, but not deposited, on the test surface. The target speed was not provided in the Study Report but was reported to be documented

in the raw data.

Equipment Calibration Procedures: The Study Report states that a calibrated sprayboom was used in the study, but

calibration procedures were not provided. According to the Study Report, the operation of the sprayboom is described in detail in Toxcon SOP No. E-042 Operation and Maintenance of the Whitmyre Application System for Pesticides (WASP). It is not certain if the equipment used in this study was consistent with the proposed use for this product. A label was not provided with the study.

Therefore, the label recommended application method is not known.

Was total deposition measured? Yes, total deposition was measured using deposition coupons. The deposition

coupons consisted of squares of alpha cellulose (3" x 3"). The coupons were backed with hexane-wiped heavy duty aluminum foil. The Study Report states that coupons were prepared according to Toxcon SOP No. M-015: Preparation of Alpha Cellulose Deposition Coupon. The coupons were present on the

wooden platforms during test substance application.

C. Sampling:

Surface Areas Sampled: Vinyl and carpet flooring sections (12" x 8") were first treated with the test

> product. Cotton glove coupons attached to wooden blocks (10" x 10") were used to sample the residues found on the vinyl and carpet flooring. The surface area of the cotton glove coupons in contact with the treated flooring materials was approximately 100 cm2. The deposition coupons consisted of 3" x 3"

squares of alpha cellulose with a surface area of 57.8 cm2.

Replicates per sampling interval: Four rounds (or sets) of triplicate glove coupons were collected for each set of

presses (2, 4, 8, 16 or 32) on either the vinyl or carpet flooring sections. A total of 30 glove coupons were generated per round (15 for vinyl flooring and 15 for carpet flooring). An overall total of 120 glove coupons were generated after all

four rounds (60 for vinyl flooring and 60 for carpet flooring).

Number of sampling intervals: Only one sampling interval was intended to collect samples. However, there was

insufficient time to complete the experiment the same day as the application. The glove coupon samples for the 32 presses on vinyl flooring and for the 2, 4, 8, 16 and 32 presses on carpet flooring were collected the day after the spray

application.

Method and Equipment: Residue deposition was determined using alpha cellulose coupons. The

> transferability of PY and PBO was determined as a function of the different number of presses on new or the same treated area using cotton glove coupons.

Sampling Procedure(s):

The deposition coupons were collected following a drying period after Deposition coupons -

application of the test product. Disposable latex gloves were worn when the coupons were handled. The coupons were folded, so that the exposed side was

on the inside, and then wrapped in hexane-wiped aluminum foil.

Cotton glove residues-Cotton glove coupons were selected instead of bare hands to eliminate the

variability introduced by using the hands of different subjects. Treated vinyl and carpet flooring sections were removed from the platforms and transported to a pre-conditioned block press room. Each treated vinyl or carpet flooring section was placed on a table. Pre-washed cotton gloves were cut into pieces approximately 130 cm2 in size, stapled to a piece of aluminum foil, and wrapped onto a flat 10 cm x 10 cm piece of wood. The cotton glove coupon/wood block was placed on a designated area of the vinyl or carpet flooring section and then an 8 kg weight was immediately placed on top of the

cotton glove coupon/wood block for a duration of 20 seconds. For the first round of presses a new area of formulation treated vinyl or carpet flooring section was used. Three replicates were used for each number of presses (2, 4, 8, 16, or 32). Following the first round of presses, a new cotton glove coupon was placed on the same area of each previously used flooring section for a repeat of exposure. This process was repeated three additional times for each section of flooring for a total of four rounds. After the cotton glove coupon was removed from the wooden block it was folded on a clean surface with the exposed side on the inside and wrapped with a piece of hexane-wiped aluminum foil.

## D. Sample Handling and Storage:

Both the cotton glove coupons and the deposition coupons were wrapped in aluminum foil and labeled with unique identifications. The deposition coupons (alpha cellulose samples) were placed in aluminum containers and the cotton glove coupons were placed in amber glass jars. According to the study protocol, all of the samples were to be placed into freezer storage ( $<10^{\circ}$ C) within 3 hours of collection. However, the transfer of the collected samples to freezer storage within the 3-hour period required was overlooked and all samples were placed into freezer storage at one time at the end of the experiment (the day after the application). All samples were stored in the dark at <-  $10^{\circ}$ C until shipped for analysis. Samples were shipped to the analytical laboratory overnight in an insulated cooler with dry ice. The samples were received by Xenos Laboratories on September 13, 2000.

## IV. ANALYTICAL METHODOLOGIES

#### A. Extraction method:

Extraction of Pyrethrin I (P-I), Cinerin I (C-I), Jasmolin I (J-I), and PBO residues was performed by sonication and mechanical shaking of the alpha cellulose and cotton glove coupons at room temperature with ethyl acetate. One extraction was performed and the ethyl acetate was taken to dryness by rotary evaporation. Two clean-up steps were required for the cotton glove coupons, including the use of a Discovery<sup>TM</sup> polyamide SPE cartridge and an Isolute silica SPE cartridge. Alpha cellulose extracts required only the Discovery<sup>TM</sup> polyamide SPE cartridge clean- up. All sample extracts were taken to dryness and made up to an appropriate volume in toluene.

**B. Detection methods:** Analysis was performed using GC/MS in the EI/SIM mode (see Table 1). The method measured three Pyrethrin esters (PYI): Pyrethrin I (P-I), Cinerin I (C-I) and Jasmolin I (J- I), and PBO. See Table 1 for specific conditions.

Table 1. Gas Chromatographic / Mass Spectrometer Conditions							
GC Column	DB-5, ~15 m x 0.25 mm ID, 0.25 mm film						
Temperatures	Inlet:						
	Initial - 120°C (hold 0.15 min)						
	Prog 1 - 120-250°C @ 200°C/min (hold 10 min)						
	Column:						
	Initial - 90°C (hold 1.5 min)						
	Prog 1 - 90-160°C @ 30oC/min						
	Prog 2 - 160-175°C @ 1.8oC/min						
	Prog 3 - 175-200°C @ 2.0oC/min						
	Prog 4 - 200-320°C @ 50oC/min (hold 15 min)						
	Transfer line: 280°C						
	Mass Spectrometer trap set temperature: 225°C						
Carrier Gas Flow Rate	~1.3 mL/min (constant)						
Mass Spectrometer Interface	direct capillary interface						
GC/MS Mode	EI/SIM mode						
Injector Split	0 min, split ON, split ratio: 10						
	0.25 min, split OFF						
	2.00 min, split ON, split ratio: 100						

Injection Volume	5.0 mL direct injection
Rate	0.4 mL/sec
Quantitating Mass Ions	PYI (all esters) = $m/z$ 123 ion
	PBO = m/z 193 ion
Approximate Retention Times	C-I ~ 17 min
	J-I ~ 20 min
	P-I ~ 21 min
	PBO ~ 23 min

#### C. Method Validation:

The analytical method was validated prior to initiation of the field phase of this study to determine the integrity and efficiency of Xenos' Analytical Method XAM-60 which was used for the analysis of the three Pyrethrin esters (PYI): Pyrethrin I (P-I), Cinerin I (C-I) and Jasmolin I (J-I) and PBO residues in/on cotton glove coupons and alpha cellulose samples.

The Study Report provides validation data for the limits of quantitation (LOQ) which were taken from Xenos report XEN00-14. Method validation recoveries were not provided in the study. However, the validated limits of quantitation (LOQ) for PYI, PY and PBO residues on alpha cellulose and cotton gloves are summarized in Table 2.

Table 2. Validated LOQs <sup>1</sup>									
Matrix	Formulation	LOQ (µg)							
	Formulation	PYI	PY	PBO					
Alpha Cellulose	10.0 μg	44.1	78.8	160					
Cotton Gloves	200 μg	0.882	1.58	3.2					

1 Validation data from Xenos report XEN00-14.

Instrument performance and calibration:

Calibration solutions were prepared from the formulation by dilution in toluene. A total of 5 concentrations were used to calibrate the system: 0.010, 0.020, 0.040, 0.075, and 0.100  $\mu g/\mu L$ . The GC/MS response was determined using the prepared calibration standards to perform a linear regression analysis. The demonstrated R2 values for PYI and PBO were 0.9733 and 0.9739, respectively, which were less than the SOP criteria of being equal to or greater than 0.98.

#### **D.** Quality Control:

Lab Recovery:

To obtain recovery and method performance data, concurrent laboratory control samples were fortified with the formulated product, prior to extraction, within the concentration ranges expected from the field samples. According to the study protocol, average laboratory recoveries in the range of 70 to 120% with a coefficient of variation of +/- 20% were considered acceptable for the study. Concurrent laboratory control samples were prepared and run for both the alpha cellulose coupons and the cotton glove coupons. Results from the laboratory fortified samples are summarized in Table 3. Since the recoveries of all but five of the laboratory fortified control samples were within the acceptance criteria of 70% to 120%, they verified method performance throughout the analytical phase of the study.

For the alpha cellulose fortified laboratory controls, one sample was fortified at the LOQ for PYI and PBO and one sample was fortified at 5X the LOQ for PYI and PBO. The recovery of the low level spike for PYI was 84% versus 85.5% at the high level spike. The recovery of the low level spike for PBO was 83.6% versus 88.5% at the high level spike. Overall average recoveries were  $84.8 \pm 1.06\%$  for PYI and  $86.1 \pm 3.46\%$  for PBO.

For the cotton glove fortified laboratory controls, there were nine samples fortified at the LOQ for PYI and PBO, five samples fortified at 2X the LOQ, six samples fortified at 5X the LOQ, five samples fortified at 10X the LOQ, and one sample each at 12.5X, 25X, and 200X the LOQ. Fortified laboratory control recoveries ranged from 65.2% to 117% for PYI and from 74.8% to 121% for PBO. Four recoveries of PYI and one recovery of PBO were outside the acceptance criteria of 70% to 120%. The overall average recoveries were  $83.6 \pm 14.1\%$  and  $92.5 \pm 12.9\%$  for PYI and PBO, respectively.

Table 3. Summary of Concurrent Laboratory Fortification Recoveries												
Matrix	Fortification Matrix Level (μg) <sup>1</sup>		No. of Samples	Average Percent Recovery (%)		Overall Average Recovery (%)		Std.	Dev.	% RSD		
	PY1	PBO	_	PYI	PBO	PYI	PBO	PYI	PBO	PYI	PBO	
Alpha	41.8	164	1	84	83.6	04.0	86.1	1.06	3.46	1.25	4.03	
Cellulose	209	820	1	85.5	88.5	84.8		1.00	3.40		4.03	
	0.836	3.28	9	92.9	99.5					16.8	14	
	1.67	6.56	5	77.3	84.9			14.1	4.1 12.9			
Cotton	4.18	16.4	6	76.6	87.2							
Glove	8.36	32.8	5	81.5	93.7	83.6	92.5					
Giove	10.5	41	1	71.8	89.2							
	20.9	82	1	76.8	89.4							
	167	656	1	102	100							

1Alpha cellulose LOQ for PYI = 41.8  $\mu$ g/sample and the LOQ for PBO = 164  $\mu$ g/sample Cotton glove LOQ for PYI = 0.836  $\mu$ g/sample and the LOQ for PBO = 3.28  $\mu$ g/sample Fortification levels are at 1X, and 5X the LOQ for alpha cellulose, and at 1X, 2X, 5X, 10X, 12.5X, 25X, and 200X the LOQ for cotton gloves.

Field Fortification:

Duplicate alpha cellulose coupon and cotton glove coupon field control samples were fortified with the formulated test product at 3X the LOQ. These field fortified control samples were exposed for the same amount of time and under the same conditions as the test samples. They were also stored and analyzed with the test samples. Field fortification results are summarized in Table 4. Alpha cellulose overall average recoveries were 70.0%  $\pm$  6.01% for PYI and 66.8%  $\pm$  7.42% for PBO. Cotton glove overall average recoveries were 115%  $\pm$  9.90% for PYI and 118%  $\pm$  4.24% for PBO.

Table 4. Summary of Field Fortification Recoveries.														
Matrix	Fortification Level (µg) 1  PYI PBO				Measured Residue (μg/sample)		Percent Recovery (%)		Overall Average Recovery (%)		e Std. Dev.		% RSD	
			PYI	PBO	PYI	PBO	PYI	PBO	PYI	PBO	PYI	PBO		
Alpha Cellulose	128	502	84 94.9	309 361	65.7 74.2	61.5 72	70	66.8	6.01	7.42	8.59	11.1		
Cotton Glove	2.56	10	2.75 3.11	11.5 12.1	108 122	115 121	115	118	9.9	4.24	8.61	3.6		

1Alpha cellulose LOQ for PYI = 41.8  $\mu$ g/sample and the LOQ for PBO = 164  $\mu$ g/sample Cotton glove LOQ for PYI = 0.836  $\mu$ g/sample and the LOQ for PBO = 3.28  $\mu$ g/sample Fortification was a 3X the LOQ for alpha cellulose and cotton gloves.

Control Samples:

One blank alpha cellulose coupon and fourteen blank cotton glove coupons were prepared for the laboratory samples. Duplicate blank alpha cellulose coupon and cotton glove coupon samples were prepared for the field samples. The blank samples were prepared by adding a volume of solvent approximately equivalent to the largest volume of solution used in fortification to alpha cellulose and cotton glove coupons. The coupons were folded with the exposed sides on the inside, wrapped in hexane-wiped foil,

and then labeled. The results for the alpha cellulose laboratory blank coupons showed detectable residue levels of PYI and PBO below the limit of quantification (LOQ). There were no detectable residues for PYI or PBO in the alpha cellulose field blank coupons. Thirteen of fourteen cotton glove laboratory blank coupons showed no detectable residues of PYI or PBO. The detectable residue in the fourteenth control sample was for PYI but it was still less than the LOQ. The results for both of the cotton glove field blank coupons showed detectable PYI and PBO recoveries, but they were below the LOQ. These results indicate the possibility of slight contamination.

Storage Stability:

The field fortified samples for alpha cellulose and cotton glove coupons were analyzed after a period of 43 and 35 days, respectively. Although the low PYI and PBO field fortification recoveries for alpha cellulose coupons could indicate that both PY and PBO might not be stable on this matrix, the Study Report stated that this was unlikely because both PY and PBO were shown to be stable on alpha cellulose during a six month stability study (XEN98-26).

#### V. RESULTS

Residues were reported for both PYI and PBO, as well as PY, which is total pyrethrin calculated from the PYI data by using a conversion factor (1.842). This conversion factor was derived from the percentages of total pyrethrins and PYI in the formulated product. Versar corrected residue data for alpha cellulose coupon field fortification recoveries below 90%. The study author did not correct for field fortification recoveries.

#### A. Alpha Cellulose and Deposition of Formulation:

The alpha cellulose coupons used to determine the application rate of the sprayboom were analyzed as part of Toxcon study 00-033-PY01 (Xenos Report XEN00-32). However, a separate set of alpha cellulose coupon quality control samples (laboratory and field blanks and fortification samples) were prepared and analyzed specific to this study. According to the Xenos Report (XEN00-32), the overall average residues were reported as  $4.01 \pm 0.812$  µg/cm2 for PY and  $8.76 \pm 1.73$  µg/cm2 for PBO. Using the field fortified QC samples specific to this study (XEN00-33), Versar corrected PYI residues for a field fortification recovery of 70.0% and PBO residues for a field fortification recovery of 66.8%. Average corrected residues for PY and PBO were  $5.73 \pm 1.16$  µg/cm2 and  $13.1 \pm 2.59$  µg/cm2, respectively. The achieved deposition rate is estimated to be 145% of the target deposition rate for PY and 167% of the target deposition rate for PBO.

#### **B.** Glove Coupon Residues

The overall average field fortification recoveries for the cotton glove coupon QC samples were 115% for PYI and 118% for PBO. Therefore, the cotton glove coupon sample residue data did not require correction for field fortification recoveries. PY is total pyrethrin calculated by using a conversion factor (1.842) derived from the percentages of total pyrethrins and PYI in the formulated product. The degree of transfer of PY and PBO residues from vinyl and carpet flooring was carried out by placing sections of cotton gloves measuring approximately 100 cm2 on the treated surfaces and applying multiple presses on a new or the same treated area. Four rounds of 2, 4, 8, 16, and 32 presses were used. The Study Report provided residue data as  $\mu g/\text{sample}$ . Versar calculated transfer residue data as  $\mu g/\text{cm2}$ . Summaries of Versar's calculated PY and PBO transfer residues resulting from multiple presses on vinyl and carpet flooring are provided in Tables 5a and 5b.

Of the 60 cotton glove coupons generated for vinyl flooring, only one glove coupon showed no detectable residues of either PY or PBO (replicate for 32 presses). For the remaining glove coupons, individual replicate transfer residues of PY ranged from 0.016  $\mu$ g/cm2 to 0.227  $\mu$ g/cm2. Individual replicate transfer residues of PBO ranged from 0.025  $\mu$ g/cm2 to 0.339  $\mu$ g/cm2. The average residues for the different number of presses for each of the four rounds ranged from 0.019  $\mu$ g/cm2  $\pm$  0.004  $\mu$ g/cm2 (4th round, after 2 presses) to 0.180  $\mu$ g/cm2  $\pm$  0.050  $\mu$ g/cm2 (1st round, after 16 presses) for PY and from 0.029  $\mu$ g/cm2  $\pm$  0.006  $\mu$ g/cm2 (4th round, after 2 presses) to 0.278  $\mu$ g/cm2  $\pm$  0.067  $\mu$ g/cm2 (1st round, after 16 presses) for PBO.

Cotton glove coupons generated for carpet flooring, showed individual replicate transfer residues of PY ranging from  $0.032~\mu g/cm2$  to  $0.895~\mu g/cm2$ . Individual replicate transfer residues of PBO ranged from  $0.074~\mu g/cm2$  to  $1.85~\mu g/cm2$ . The average residues for the different number of presses for each of the four rounds ranged from  $0.041~\mu g/cm2 \pm 0.008~\mu g/cm2$  (4th round, after 2 presses) to  $0.825~\mu g/cm2 \pm 0.065~\mu g/cm2$  (1st round, after 32 presses) for PY and from  $0.091~\mu g/cm2 \pm 0.013~\mu g/cm2$  (4th round, after 2 presses) to  $1.073~\mu g/cm2 \pm 0.133~\mu g/cm2$  (1st round, after 32 presses) for PBO.

The percent of residue on the cotton glove coupons after contact with treated vinyl and carpet flooring surfaces was calculated as the ratio of the amount of residue present on the glove coupons divided by the average corrected residue found on the alpha cellulose coupons (5.73  $\mu$ g/cm2 for PY and 13.1  $\mu$ g/cm2 for PBO). Versar calculated the average percentages of PY and PBO residues transferred from vinyl and carpet surfaces for each of the total number of multiple presses for the four rounds of presses and for each of the individual presses for the four rounds. These percentages were provided in Tables 5a and 5b (per total presses). Tables 5a and 5b also provide the percent of residue transferred per press.

#### VI. CONCLUSION

The transfer of PY and PBO residues from treated vinyl and carpet flooring to cotton glove coupons following multiple presses on new or the same treated area was measured. The results of this study indicated that for vinyl and carpet flooring, the per press residues of both PY and PBO transferred from the flooring surface to the cotton glove coupons decreased as the number of presses increased. For vinyl, the percent of PY transferred to the cotton glove coupons was greater than the percent of PBO transferred. For carpet, the percent of PY and PBO transferred to the cotton glove coupons was similar.

#### LIMITATIONS OF THE STUDY:

The protocol provided with the study along with OPPTS Series 875 Part B, Guideline 875.2300: Indoor Surface Residue Dissipation, Postapplication and Part C Guidelines were used to review the study. Overall, the majority of the procedures performed and the quality of the data generated in this study conformed to the criteria set forth in the protocol and guidelines. However, certain issues of concern were noted:

- A specific application rate was not provided in the Study Report. Application was based on a target deposition rate determined in another study.
- The test product was not identified and a label was not provided.
- Calibration procedures for the application equipment were not provided in the Study Report.
- Only one sampling interval was intended to collect samples. However, there was insufficient time to complete the experiment the same day as the application and the remainder of the samples were collected on the day after the application.
- The requirement for the transfer of collected samples to freezer storage within the 3-hour period was overlooked and all samples were placed into freezer storage at one time at the end of the experiment (the day after the application).
- Only one field fortification level with duplicate field fortified control samples were prepared for alpha cellulose and cotton glove coupons.
- Alpha cellulose coupons used to determine the application rate of the sprayboom were analyzed as part of another study (Xenos Report XEN00-32; Vol. 8). However, alpha cellulose coupon QC samples were prepared and analyzed for specifically for this study. The field fortification recoveries for both PY and PBO were below 90% and the alpha cellulose coupon residue data were corrected accordingly.
- Surface sampling was not conducted in conjunction with air sampling to establish a dissipation curve.

Table 5a. Summary of Pyrethrin and PBO Weighted Press Results on Vinyl Flooring																	
Treated Surface	Rolling		Glove Surface Area	Average Residue by # of Presses (µg/cm2) <sup>c,d</sup>			Standard			% of Application by # of Presses <sup>f</sup>		Average Residue per Press (µg/cm2) <sup>g</sup>		% of Application per Press <sup>f</sup>			
			(cm2) <sup>b</sup>	PYI	PY <sup>e</sup>	PBO	PYI	PYe	PBO	PY	PBO	PY	PBO	PY	PBO		
Vinyl		2	100	0.020	0.037	0.053	0.004	0.007	0.010	0.645	0.404	0.018	0.026	0.323	0.202		
, .		4	100	0.031	0.057	0.093	0.007	0.013	0.021	1.00	0.709	0.014	0.023	0.250	0.177		
	1	8	100	0.059	0.108	0.160	0.004	0.007	0.010	1.88	1.22	0.014	0.020	0.236	0.153		
		16	100	0.098	0.180	0.278	0.027	0.050	0.067	3.15	2.12	0.011	0.017	0.197	0.133		
		32	100	0.050	0.092	0.140	0.011	0.021	0.029	1.61	1.07	0.003	0.004	0.050	0.033		
		2	100	0.022	0.041	0.061	0.004	0.007	0.009	0.719	0.469	0.021	0.031	0.360	0.235		
		4	100	0.024	0.044	0.064	0.003	0.005	0.007	0.776	0.491	0.011	0.016	0.194	0.123		
	2	8	100	0.048	0.087	0.127	0.004	0.007	0.007	1.53	0.972	0.011	0.016	0.191	0.122		
		16	100	0.069	0.128	0.194	0.015	0.028	0.048	2.23	1.48	0.008	0.012	0.139	0.092		
		32	100	0.050	0.091	0.130	0.010	0.019	0.019	1.60	0.995	0.003	0.004	0.050	0.031		
		2	100	0.011h	0.020h	0.034h	0.0006	0.011	0.016	0.352	0.259	0.010	0.017	0.176	0.130		
		4	100	0.019	0.035	0.056	0.001	0.001	0.0005	0.609	0.424	0.009	0.014	0.152	0.106		
	3	8	100	0.042	0.078	0.120	0.011	0.020	0.028	1.36	0.914	0.010	0.015	0.170	0.114		
				16	100	0.077	0.142	0.204	0.024	0.045	0.057	2.48	1.56	0.009	0.013	0.155	0.097
		32	100	0.038	0.070	0.091	0.010	0.019	0.020	1.22	0.694	0.002	0.003	0.038	0.022		
		2	100	0.010	0.019	0.029	0.002	0.004	0.006	0.334	0.225	0.010	0.015	0.167	0.112		
		4	100	0.018	0.033	0.050	0.003	0.005	0.007	0.579	0.381	0.008	0.012	0.145	0.095		
	4	8	100	0.034	0.063	0.090	0.008	0.015	0.025	1.10	0.688	0.008	0.011	0.137	0.086		
		16	100	0.067	0.123	0.168	0.012	0.022	0.027	2.15	1.28	0.008	0.011	0.134	0.080		
		32	100	0.026	0.048	0.063	0.002	0.004	0.002	0.833	0.478	0.001	0.002	0.026	0.015		

- a For the first round of presses a new area of formulation treated flooring section was used. Following the first round of presses, a new cotton glove coupon was placed on the same area of each previously used flooring section for a repeat of exposure. This process was repeated three additional times for each section of flooring for a total of four rounds.
- b Surface area of the glove coupon was based on the 10 cm X 10 cm area of coupon which was in contact with the floor surface.
- c No correction needed since field recoveries were above 90% (PYI=115% and PBO=118%).
- d Average of triplicate samples and converted from μg/sample to μg/cm2 based on glove coupon surface area.
- e PY is total pyrethrin calculated by using a conversion factor (1.842) derived from the percentages of total pyrethrins and PYI in the formulated product.
- f Calculated as the ratio of the amount of residue present on the glove coupon divided by the average residue found on the alpha cellulose coupons (5.73  $\mu$ g/cm2 for PY and 13.1  $\mu$ g/cm2 for PBO).
- g Calculated by dividing the average residue by number of presses by the number of presses.
- h Residues were <LOQ for one replicate. Averages and standard deviations were calculated using ½ LOQ (1.58 μg for PY and 3.2 μg for PBO).

Table 5b. Summary of Pyrethrin and PBO Weighted Press Results on Carpet Flooring																
Treated Surface	Rounds <sup>a</sup>	# of Presses	# of Glove		Average Residue by #  of Presses (µg/cm2) <sup>c,d</sup>			Standard Deviation by # of Presses (µg/cm2)			% of Application		Average Residue per Press (µg/cm2) <sup>g</sup>		% of Application per Press <sup>f</sup>	
			(cm2) <sup>b</sup>	PYI	$PY^{e}$	PBO	PYI	PY <sup>e</sup>	PBO	PY	PBO	PY	PBO	PY	PBO	
		2	100	0.042	0.077	0.165	0.007	0.013	0.027	1.34	1.26	0.038	0.082	0.670	0.628	
		4	100	0.091	0.167	0.370	0.023	0.042	0.102	2.91	2.83	0.042	0.093	0.729	0.707	
	1	8	100	0.140	0.258	0.569	0.021	0.039	0.087	4.51	4.34	0.032	0.071	0.564	0.543	
		16	100	0.239	0.441	0.914	0.060	0.110	0.271	7.69	6.97	0.028	0.057	0.481	0.436	
		32	100	0.448	0.825	1.703	0.035	0.065	0.133	14.4	13.0	0.026	0.053	0.450	0.406	
		2	100	0.031	0.057	0.113	0.006	0.011	0.018	0.989	0.859	0.028	0.056	0.495	0.430	
		4	100	0.056	0.103	0.231	0.008	0.015	0.044	1.80	1.76	0.026	0.058	0.450	0.440	
	2	8	100	0.128	0.236	0.491	0.051	0.094	0.178	4.12	3.75	0.030	0.061	0.515	0.469	
		16	100	0.169	0.312	0.653	0.045	0.083	0.175	5.44	4.98	0.019	0.041	0.340	0.311	
Carpet		32	100	0.340	0.626	1.290	0.076	0.140	0.238	10.9	9.85	0.020	0.040	0.342	0.308	
Carper		2	100	0.023	0.043	0.094	0.004	0.008	0.020	0.750	0.714	0.021	0.047	0.375	0.357	
		4	100	0.050	0.092	0.199	0.007	0.014	0.034	1.61	1.52	0.023	0.050	0.403	0.379	
	3	8	100	0.085	0.156	0.341	0.012	0.023	0.045	2.72	2.61	0.019	0.043	0.340	0.326	
			16	100	0.150	0.276	0.626	0.024	0.044	0.126	4.82	4.78	0.017	0.039	0.301	0.299
		32	100	0.225	0.415	0.914	0.026	0.049	0.134	7.24	6.98	0.013	0.029	0.226	0.218	
		2	100	0.022	0.041	0.091	0.004	0.008	0.013	0.719	0.693	0.021	0.045	0.360	0.347	
		4	100	0.047	0.086	0.190	0.006	0.011	0.032	1.50	1.45	0.022	0.047	0.376	0.362	
	4	8	100	0.090	0.165	0.374	0.009	0.016	0.029	2.89	2.85	0.021	0.047	0.361	0.357	
		16	100	0.138	0.255	0.552	0.035	0.064	0.129	4.45	4.22	0.016	0.035	0.278	0.264	
		32	100	0.238	0.438	0.871	0.030	0.055	0.099	7.64	6.65	0.014	0.027	0.239	0.208	

- For the first round of presses a new area of formulation treated flooring section was used. Following the first round of presses, a new cotton glove coupon was placed on the same area of each previously used flooring section for a repeat of exposure. This process was repeated three additional times for each section of flooring for a total of four rounds.
- b Surface area of the glove coupon was based on the 10 cm X 10 cm area of coupon which was in contact with the floor surface.
- c No correction needed since field recoveries were above 90% (PYI=115% and PBO=118%).
- d Average of triplicate samples and converted from μg/sample to μg/cm2 based on glove coupon surface area.
- e PY is total pyrethrin calculated by using a conversion factor (1.842) derived from the percentages of total pyrethrins and PYI in the formulated product.
- Calculated as the ratio of the amount of residue present on the glove coupon divided by the average residue found on the alpha cellulose coupons (5.73  $\mu$ g/cm2 for PY and 13.1  $\mu$ g/cm2 for PBO).
- g Calculated by dividing the average residue by number of presses by the number of presses.

Name: Evaluator Occupational Exposure Assessment Section	Name:	Peer Reviewer Occupational Exposure Assessment Section
Date	Date	
Name: Head, Occupational Exposure Assessment Section	on	

# APPENDIX A

Compliance Checklist for "Measurement of Transfer of Pyrethrin and Piperonyl Butoxide Residues from Vinyl or Carpet Flooring Treated with a Fogger Formulation to Glove Coupons Following Multiple Presses" Compliance Checklist for "Measurement of Transfer of Pyrethrin and Piperonyl Butoxide Residues from Vinyl or Carpet Flooring Treated with a Fogger Formulation to Glove Coupons Following Multiple Presses"

## GUIDELINE 875,2300 INDOOR SURFACE RESIDUE DISSIPATION POSTAPPLICATION

- 1. The test substance must be the typical end use product of the active ingredient. It is unclear if this criterion was met. The test product was an unidentified product and no label was provided.
- 2. The production of metabolites, breakdown products, or the presence of contaminants of potential toxicologic concern, should be considered on a case-by-case basis. This criterion does not appear to apply to this study.
- 3. Indoor surface residue studies should be conducted under ambient conditions similar to those encountered during the intended use season, and should represent reasonable worst case conditions. This criterion was met.
- 4. Ambient conditions (i.e., temperature, barometric pressure, ventilation) should be monitored. This criterion was mostly met. The target conditions were identified and apparently met, but monitoring data were not provided in the Study Report.
- 5. The end use product should be applied by the application method recommended on the label. Information that verifies that the application equipment (e.g., sprayer) was properly calibrated should be included. It is not certain if these criteria were met completely. Information was provided verifying the calibration of the application equipment. However, it is uncertain if the test product was applied by the application method recommended on the label because the label for the test product was not provided with the study.
- 6. The application rate used in the study should be provided and should be the maximum rate specified on the label. However, monitoring following application at a typical application rate is more appropriate in certain cases. This criterion was not met. Application was based on a target deposition rate, determined in another study.
- 7. *If multiple applications are made, the minimum allowable interval between applications should be used.* This criterion does not apply to this study. There was only one application.
- 8. Indoor surface residue (ISR) data should be collected from several different types of media (e.g., carpeting, hard surface flooring, counter tops, or other relevant materials). This criterion was met. Indoor surface residues were collected from vinyl and carpet flooring. The objective of the study was to determine glove coupon press residue from contact with treated vinyl and carpet flooring sections.
- 9. Sampling should be sufficient to characterize the dissipation mechanisms of the compound (e.g., three half-lives or 72 hours after application, unless the compound has been found to fully dissipate in less time; for more persistent pesticides, longer sampling periods may be necessary). Sampling intervals may be relatively short in the beginning and lengthen as the study progresses. Background samples should be collected before application of the test substance occurs. This criterion does not apply to this study.
- 10. Triplicate, randomly collected samples should be collected at each sampling interval for each surface type. This criterion was mostly met. Triplicate samples were collected. However, the randomness of the sample collection is not certain.
- 11. Samples should be collected using a suitable methodology (e.g., California Cloth Roller, Polyurethane Roller, Drag Sled, Coupons, Wipe Samples, Hand Press, vacuum cleaners for dust and debris, etc.) for indoor surfaces. This criterion was met. Samples were collected using cotton glove coupons.

- 12. Surface sampling should be conducted in conjunction with air sampling. Enough duplicate air samples should be taken in a room to establish a dissipation curve. This criterion was not met.
- 13. Samples should be stored in a manner that will minimize deterioration and loss of analytes between collection and analysis. Information on storage stability should be provided. This criterion was met. Field fortification samples were analyzed after a period of 43 days for alpha cellulose coupons and 35 days for cotton glove coupons to ensure storage stability.
- 14. Validated analytical methods of sufficient sensitivity are needed. Information on method efficiency (residue recovery), and limit of quantitation (LOQ) should be provided. This criterion was met.
- 15. Information on recovery samples must be included in the study report. A complete set of field recoveries should consist of at least one blank control sample and three or more each of a low-level and high-level fortification. These fortifications should be in the range of anticipated residue levels in the field study.

  This criterion was partially met. Blank control samples and field fortification samples were included in the study, but only duplicate replicates of a single fortification level were reported.
- 16. Raw residue data must be corrected if appropriate recovery values are less than 90 percent. This criterion was not met. Alpha cellulose coupon residue data were not corrected for an overall average field fortification recovery of 70.0% for PYI and 66.8% for PBO. Cotton glove coupon residue data did not require correction.
- 17. Indoor surface residues should be reported as mg per m2 or cm2 of surface sampled. Distributional data should be reported, to the extent possible. These criteria were partially met. Residues from cotton glove press samples were reported as ·g/sample and ng/cm2.
- 18. Reported residue dissipation data in conjunction with toxicity data should be sufficient to support the determination of a reentry interval. This criterion does not apply to this study.